

(19) Japan Patent Agency (JP)

(10) **Public Patent bulletin**

(11) Public Patent Application Number

**Sho 63 - 86738**

(43) Date of being Public Showa 63 (1988), 18<sup>th</sup> April

(51) Int. Cl<sup>4</sup>. Discrimination Internal Arrangement

	Number	Number
C 08 L 25/06	LED	7445 - 4J
B 41 M 5/26		V - 7265 - 2H
C 08 L 71/04	LQP	8016 - 4J
G 02 B 1/04		7915 - 2H
G 11 B 7/24		Z - 8421 - 5D

Investigations of Claims Nil

Number of Inventions 1

(Total 5 Pages (Japanese))

(72) Inventor

Tachiue Yoshiharu

Oosaka fu, Oosaka shi,

Takatsuki shi,

Tsukahara, 2 cho me,

10 ban, 1 go.

Sumitomo Kagaku Kogyo

Kabushiki Kaisha Nai

(Internal to Sumitomo

Chemical Industries

Ltd.)

(72) Inventor

Nakarai Toyoake

Oosaka fu, Oosaka shi,

Takatsuki shi,

Tsukahara, 2 cho me,

10 ban, 1 go. .

Sumitomo Kagaku Kogyo

Kabushiki Kaisha Nai

(Internal to Sumitomo

Chemical Industries

Ltd.)

(71) Patent Applicant

Sumitomo Kagaku Kogyo

Kabushiki Kaisha

(Sumitomo Chemical

Industries Ltd.)

Oosaka fu, Oosaka shi,

Higashi ku, Kitahama,

5 cho me, 15 ban chi..

(74) Representative Patent Attorney Moroichi Komori

(Besides one more name)

## A detailed description

### 1. Title of the invention

Resin Composite for Optical Disk Baseboard.

## 2. Scope of patent claims

- (1) The resin composite for optical disk baseboard that is formed from 20 ~ 45 weight % of polyphenylene oxide that is formed by polymerizing styrene under the presence of polyphenylene oxide resin, and 80 ~ 55 weight % of polystyrene resin.
- (2) The resin composite for optical disk baseboard described above in claim number 1 in which styrene resin of polyphenylene oxide resin is filtered before carrying out polymerization.

## 3. Detailed description of the invention

### <Industrial applicability>

The present invention relates to a resin composite. In further details, the present invention relates to the resin composite for optical disk baseboard that is formed from polyphenylene oxide resin and polystyrene resin.

### <Techniques of the past>

Recently, the resin composite showing excellent optical

isotropy (namely, low birefringence (double refraction)) formed from 44 ~ 40 parts by weight of polyphenylene oxide resin formed by blending polyphenylene oxide resin and poly methylene resin, and 66 ~ 60 parts by weight of polystyrene resin, has been presented (refer to American patent 4378066). This resin composite has excellent optical isotropy, in addition to possessing the characteristic of not losing this excellent optical isotropy even by deformation taking place due to stretching, and thus, it is being used in lenses or optical disks used as information recording medium.

**<Problems the invention solves>**

The optical disk that is formed by using the resin composite mentioned above in the baseboard material, has satisfactory optical isotropy, however, it has the problem of not having sufficiently high carrier - noise ratio (called as C / N ratio) at the time of recording and regeneration.

The present invention aims at offering a resin composite for optical disk baseboard that is appropriate to be used in the manufacture of optical disk possessing sufficiently high C / N ratio at the time of recording and regeneration.

**<Method to solve the problems>**

The authors of the present invention carried out an earnest research regarding the resin composite for optical disk baseboard formed from polyphenylene oxide resin and polystyrene resin. As a result of this, they showed that the resin composite formed from polyphenylene oxide resin that is obtained by polymerizing styrene under the presence of polyphenylene oxide resin, and polystyrene resin is excellent to be used as the resin composite for optical disk baseboard. Based on this knowledge, the present invention was completed. Namely, the present invention relates to the resin composite for optical disk baseboard that is formed from 20 ~ 45 weight %

of polyphenylene oxide that is formed by polymerizing styrene under the presence of polyphenylene oxide resin, and 80 ~ 56 weight % of polystyrene resin.

As regards the examples of polyphenylene oxide resin used in the present invention, poly (2, 6 - di methyl - 1, 4 - phenylene oxide), poly (2, 6 - di ethyl - 1, 4 - phenylene oxide), poly (2 - methyl - 6 - ethyl - 1, 4 - phenylene oxide), poly (2 - methyl - 6 - propyl - 1, 4 - phenylene oxide), poly (2, 6 - di propyl - 1, 4 - phenylene oxide), poly (2 - ethyl - 6 - propyl - 1, 4 - phenylene oxide), poly (2, 3, 6 - tri methyl - 1, 4 - phenylene oxide) etc. can be given. The most desired polyphenylene oxide resin is poly (2, 6 - di methyl - 1, 4 - phenylene oxide). These polyphenylene oxide resins are generally manufactured by oxidative polymerization of the corresponding phenol derivative. Their manufacturing method has been described in Patent number Sho 36 - 13692, Sho 47 - 36518 and Sho 60 - 46129.

The resin composite of the present invention is obtained by polymerizing styrene under the presence of dissolved polyphenylene oxide resin using the solution formed by dissolving polyphenylene oxide resin in styrene. Here, as regards the mixing proportion of polyphenylene oxide resin and styrene, proportion of polyphenylene oxide resin in the resin composite obtained by polymerization should be 20 ~ 45 weight % and proportion of polystyrene resin should be 80 ~ 55 weight %. Desirably, proportion of polyphenylene oxide resin and polystyrene resin should be 34 ~ 40 weight % and 66 ~ 60 weight % respectively. In the case of proportion of polyphenylene oxide resin being less 20 weight % and more than 45 weight %, namely, in the case of proportion of polystyrene resin being more than 80 weight % and less than 55 weight %, birefringence (double refraction) does not lower sufficiently and C / N ratio does not increase up to the satisfactory level which is not desired. In order to make the processes of



preparation and filtration of styrene solution of polyphenylene oxide resin mentioned below, still easier, quantity of styrene used in the dissolution of polyphenylene resin should be more than the proportion mentioned above. In this case, excess quantity of styrene is removed out of the system before or during the polymerization, such that (proportion of) each component resin present in the resin composite obtained after polymerization becomes the proportion mentioned above. Moreover, the resin composite of the present invention not only includes homo polymer of polyphenylene oxide resin and polystyrene resin respectively, but it also includes graft copolymer formed from both these. In the case of containing graft copolymer, its main is polyphenylene oxide resin and branch is polystyrene resin and moreover, within the range of not losing the desired property of optical isotropy, a part of styrene can be substituted by other vinyl unimers that are copolymerizable with styrene

such as chloro styrene,  $\alpha$  - methyl styrene.

Styrene solution of the polyphenylene oxide resin is desired to be prepared at the temperature of approximately 90 ~ 130°C in order to obtain more homogeneous solution. Moreover, the solution mentioned above is desired to be filtered before carrying out polymerization in order to remove foreign substance such as dust. Temperature at the time of filtration is desired to be maintained at the temperature at which the solution mentioned above is prepared, or at the temperature more than that, in order to lower the viscosity of the solution mentioned above. As regards the filtration material used at the time of filtration, material that can remove the foreign substance such as dust etc. of the order of several  $\mu\text{m}$  and as regards such filtration material, filter paper, bulb, filter cloth, sintered metal, sintered material of metal ---, ceramic filtration material etc. can be given and the desired filtration material is filter paper and sintered

material of metal ----. As regards the filtration device, batch type filters such as pressure filter, elevated pressure type filter, vacuum filter, or continuous type filter are used.

As regards the polymerization method of styrene that is carried out under the presence of polyphenylene oxide resin, different methods generally used in the polymerization of styrene such as ion polymerization method, radical polymerization method etc. can be given. The desired polymerization method is radical polymerization method including heat polymerization. Process-wise any method among bulk polymerization method, suspension polymerization method, emulsion polymerization method, can be used, however, bulk polymerization method is desired to be used.

In the case of radical polymerization method, radical initiators generally used in polymerization of styrene such as organic peroxides such as benzoyl peroxide, di

cumyl peroxide, lauroyl peroxide, cumene hydro peroxide etc., azo compounds such as azo bis iso butylonitrile, 2 - phenyl azo - 2, 6 - di methyl - 4 - methoxy valeonitrile etc. can be used. The quantity of addition of these should be within the range from 0.01 ~ 10 weight %, desirably, 0.1 ~ 2 weight %, with respect to styrene. Moreover, polymerization can be initiated by irradiation of energy rays such as X rays, electron rays, ultraviolet rays etc. or simply by heating, without using these radical initiators. Polymerization temperature should be within the range from approximately 80 ~ 300°C, desirably, within the range from 100 ~ 270°C. Range of polymerization time differs depending on the method of polymerization initiation used in polymerization and polymerization temperature due to which it cannot be generalized, however, it is generally within the range from 10 minutes ~ 20 minutes.

Moreover, the resin composite of the present invention

thus obtained is molded into optical disk baseboard by injection molding or compression molding and different types of optical disks can be formed by forming information recording element formed from organic compound, inorganic compound or metal etc. on the baseboard by vapor deposition, sputtering or coating. As regards these methods, method of forming pit by irradiation of laser beam, method of changing optical properties by heat, opto-magnetic method in which Faraday effect or Carl (?) effect is used, etc. can be given. These methods have been described in detail in Patent number Sho 58 - 90986, Sho 57 - 109696 and Sho 61 - 16047. The resin composite of the present invention is particularly appropriate to be used in opto-magnetic system.

#### **<Effect>**

The reasons for the resin composite of the present invention (called as polymerization method resin composite) possessing exceptionally excellent properties

as optical disk baseboard, are as follows. Namely, (1) regarding the mixing conditions of both the component resins occurring in the resin composite, the polymerization method resin composite is mixed more homogeneously than the composite obtained by blending the resins (called as blend method resin composite), (2) regarding the condition of removal of foreign substances such as dust having size of the order of several  $\mu\text{m}$ , with respect to the resin composite for optical disk baseboard, solution of polymer having low viscosity formed by dissolving polyphenylene oxide resin in styrene monomer, can be extremely easily filtered, as compared to the blend method resin composite that is extremely difficult to filter due to its high viscosity, and thus, polymerization method resin composite satisfies this condition, and these are the characteristics of the manufacturing method of polymerization method resin composite.

#### **<Practical examples>**

The present invention has been explained below in further details with the help of practical examples. However, the present invention is not restricted only to these practical examples. Parts occurring in the practical examples means parts by weight. Intrinsic viscosity  $[\eta]$  is measured over chloroform solution at 25°C. Moreover, C / N ratio is the value measured by using spectrum analyzer 358A of Hewlett Packard company under the conditions of no difference effect (?) optics system and band width of scanning filter of 30 KHz. Birefringence (double refraction) is the value measured over the circular plate having diameter of 50 mm and thickness of 1 mm molded by miniature type injection molding machine (Sumitomo Jukikai (Sumitomo Heavy Machinery) make Minimat (??) at 60°C, using eripinmeter (?) under the conditions of output of He - N laser of 5 mW and single base. Quantity of foreign substance such as dust etc. is determined by the method in which the resin composite is dissolved in

clean chloroform with the concentration of 5 weight % and laser light having optical path length of 1 cm (?) is radiated in the cell on the obtained solution and the radiated spots are counted. Evaluation was carried out in 3 step given below.

A: less than 8 units

B: 4 ~ 20 units

C: more than 21 units

#### **Practical example 1**

##### **(Preparation of resin composite)**

37 parts of poly (2, 6 - di methyl - 1, 4 - phenylene oxide) ( $[\eta] = 0.51 \text{ dl/g}$ ) and 0.2 parts of cumene hydro peroxide were dissolved in 63 parts of styrene at  $90^{\circ}\text{C}$  after which Nippon --- (Japan ---) company make stainless --- filter Naslon (?)<sup>®</sup> NF - 03 was used in the filtration material and it was filtered under the elevated pressure of  $20 \text{ kg/cm}^2$  and styrene solution of poly (2, 6 - di methyl - 1, 4 - phenylene oxide) was obtained. This solution was



taken in a high viscosity reactor equipped with biaxial extruder and uniaxial extruder for injection and polymerization was carried out at room temperature under elevated pressure of 4 kg/cm<sup>2</sup>G simultaneously while increasing the temperature gradually from 100°C to 270°C for 1 hour and then, extrusion was carried out by the extruder and the resin composite was obtained as pellet. Regarding this resin composite, as a result of analysis of composition using analysis of styrene by gas chromatography and infrared spectrum, it was understood that the taken styrene was essentially completely converted into polystyrene. Moreover,  $[\eta]$  of the resin composite, quantity of foreign substance and birefringence (double refraction) have been shown in table 1.

(Preparation of opto-magnetic recording type optical disk)

The resin composite pellet mentioned above was

injection molded at 280°C and optical disk baseboard having diameter of 180 mm and thickness of 1.2 mm was prepared. According to the method described in Patent number Sho 61 - 16047, a film of terbium / iron having thickness of approximately 1.0  $\mu\text{m}$  was formed on this baseboard and opto-magnetic recording type optical disk was prepared. C / N ratio of this optical disk has been shown in table 1.

**Practical examples 2 ~ 3, comparative example 1 ~ 2**

The proportion of poly (2, 6 - di methyl - 1, 4 - phenylene oxide) and styrene was changed to the proportion of poly (2, 6 - di methyl - 1, 4 - phenylene oxide) and styrene given in table 1. Other than this change, preparation of resin composite and preparation of opto-magnetic recording type optical disk were carried out.  $[\eta]$  of the resin composite obtained here, quantity of foreign substance, birefringence (double refraction) and C / N ratio of the optical disk have been given in table

1.

### **Comparative example 3**

(Preparation of resin composite: Preparation as per American Patent 4878065)

37 parts of poly (2, 6 - di methyl - 1, 4 - phenylene oxide) ( $[\eta] = 0.51 \text{ dl/g}$ ) and 63 parts of polystyrene (Nippon Polystyrene (Japan polystyrene) (company) Esprite® Gr) were extruded by biaxial extruder and pellet of the resin composite was obtained. Its  $[\eta]$ , quantity of foreign substance and birefringence (double refraction) have been given in table 1.

(Preparation of opto-magnetic recording type optical disk)

The resin composite mentioned above was processed similar to practical example 1 and opto-magnetic recording type optical disk was prepared. Its C / N ratio has been given in table 1.

**Table 1**

Number	Composition of resin composite (weight %)		[ $\eta$ ] (dl/g)	Quant ity of forei gn subst ance	Birefring ence (double refractio n) (single) (nm)	C/ N rat io (dB )
	Poly (2, 6 - di methyl - 1, 4 - phenylene oxide)	Polys tyren e				
Practi cal exampl e 1	37	63	0.5 4	A	8	48
" 2	32	48	0.6 8	A	12	42
" 3	48	57	0.4 5	A	15	39
Compar ative exampl e 1	15	36	0.7 1	A	38	24
" 2	47	58	0.3	A	31	26

			8			
" 3	37	63	0.6	C	14	16
			1			

**<Effect / result of the invention>**

If the present invention is used, then the resin composite formed from a specific proportion of polyphenylene oxide that is obtained by polymerizing styrene under the presence of polyphenylene oxide resin, and polystyrene resin, can be used in optical disk baseboard, and thus, optical disk baseboard having excellent optical isotropy and sufficiently high C / N ratio at the time of recording and regeneration, can be obtained. As shown in table 1, optical disk obtained from the resin composite of the present invention (practical example 1 ~ 3) has satisfactory birefringence (double refraction) and C / N ratio, as compared to the optical disk obtained from polymerization method resin composite (comparative example 1 ~ 2) and the optical disk obtained

from blend method resin composite (comparative example 3), that are outside the composition range of the present invention. As mentioned above, resin composite of the present invention has low birefringence (double refraction) and low mixing of foreign substance and moreover, gives high C / N ratio at the time of processing it into optical disk and therefore, its industrial value is very high.